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Thermal and Chemical Characterization of Composite Materials
(MSFC Center Director's Discretionary Fund Final Report, Project No. ED36–18)

D.C. Stanley and T.L. Huff
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December 2003

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LIST OF ACRONYMS

DSC	differential scanning calorimeter
DTA	differential thermal analyzer
FTIR	Fourier transform infrared (spectroscopy)
MSFC	Marshall Space Flight Center
NCFI	North Carolina Foam Industries
TGA	thermogravimetric analyzer

TECHNICAL MEMORANDUM

THERMAL AND CHEMICAL CHARACTERIZATION OF COMPOSITE MATERIALS (MSFC Center Director's Discretionary Fund Final Report, Project No. ED36–18)

1. PURPOSE

The purpose of this research effort was to (1) provide a concise and well-defined property profile of current and developing composite materials using thermal and chemical characterization techniques and (2) optimize analytical testing requirements of materials.

This effort applied a diverse array of methodologies to ascertain composite material properties. Often, a single method or technique will provide useful, but nonetheless incomplete, information on material composition and/or behavior. To more completely understand and predict material properties, a broad-based analytical approach is required. By developing a database of information comprised of both thermal and chemical properties, material behavior under varying conditions may be better understood. This is even more important in the aerospace community, where new composite materials and those in the development stage have little reference data. For example, Fourier transform infrared (FTIR) spectroscopy spectral databases available for identification of vapor phase spectra, such as those generated during experiments, generally refer to well-defined chemical compounds. Because this method renders a unique thermal decomposition spectral pattern, even larger, more diverse databases, such as those found in solid and liquid phase FTIR spectroscopy libraries, cannot be used. By combining this and other available methodologies, a database specifically for new materials and materials being developed at Marshall Space Flight Center (MSFC) can be generated. In addition, characterizing materials using this approach will be extremely useful in the verification of materials and identification of anomalies in NASA-wide investigations.

2. BACKGROUND

Past efforts have primarily focused on the chemical structure of materials using FTIR spectroscopy techniques. A users library was established in 1997 and contains the chemical identification of some composites, polymers, ceramics, plastics, and rubber. MSFC is involved in the development of innovative hardware, which increasingly incorporates composites, polymers, ceramics, and other organic compounds. However, databases and characterization of these compounds are limited.

In addition, the bomb calorimeter, thermogravimetric analyzer (TGA)/FTIR spectrometer, differential thermal analyzer (DTA), differential scanning calorimeter (DSC), and stand-alone TGA have been utilized to characterize materials. However, the materials were characterized using only one of the instruments. No data were obtained for one material from all six of the listed instruments.

Programmatic support using the methodologies described above have included FASTRAC (MCC–1), X–33, VCD Flight Experiment, Space Shuttle, Space Station, BUNDLE, and Aerogel.

Efforts in the Chemistry Group focused primarily on material chemical composition using FTIR and TGA/FTIR spectroscopy methodologies. Use of the TGA in tandem with the FTIR spectrometer allows for the qualitative and quantitative determination of gaseous combustion products evolved from nonmetallic materials during heating. Thus, not only are the thermal properties of a material examined but the specific chemical species evolved during the thermal decomposition are identified. Using this technique, diverse organic species have recently been analyzed, including composites, plastics, rubber, fiberglass epoxy resins, polycarbonates, silicones, and fluorocarbons.

The TGA is used for nonmetallic material thermal decomposition studies, and when coupled with the FTIR spectrometer, provide chemical characterization of the evolving gases. The DTA and DSC provide melting point data for metallic and nonmetallic materials, while the DSC provides increased sensitivity for the study of other thermal properties, including heat of fusion and glass transition properties.

3. APPROACH

The approach taken includes the following three steps:

(1) Select materials for analysis—sample types include various epoxy resins, polyurethane foams, and thermosetting materials, including rubber.

(2) Develop a material matrix using existing thermal and chemical instrumentation in the Chemistry Branch.

(3) Analyze materials utilizing thermal analysis systems consisting of DSC, DTA, and TGA. In addition, the use of bomb calorimetry will be employed to measure material heat of combustion properties. Chemical characterization was achieved using the FTIR spectrometer alone and in combination with the TGA.

Figures 1–4 depict the curves obtained from the FTIR spectrometer, DSC, DTA, and TGA/FTIR spectrometer for one of the materials analyzed—North Carolina Foam Industries (NCFI) foam.

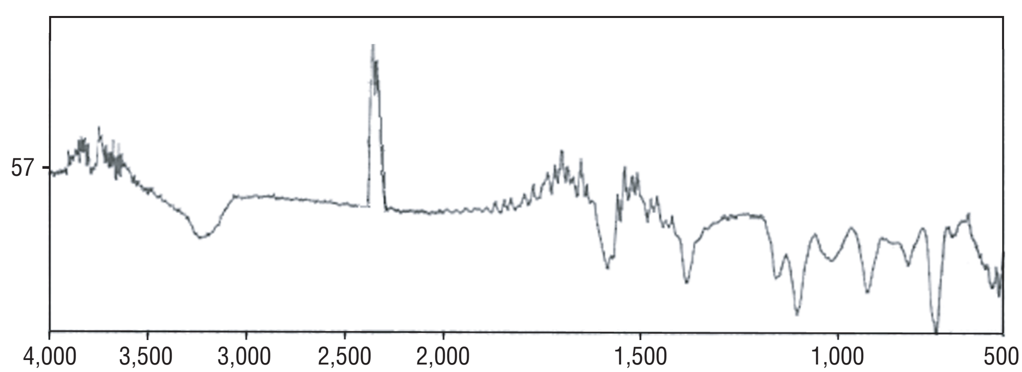

Figure 1. FTIR spectrometer spectrum of NCFI foam.

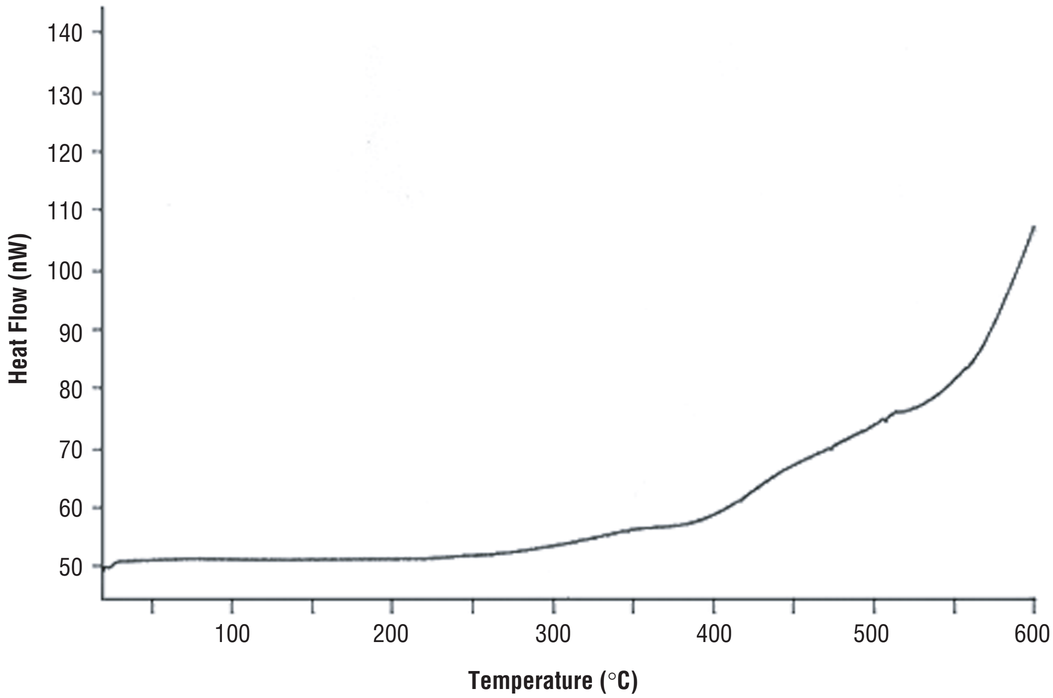

Figure 2. DSC curve of NCFI foam.

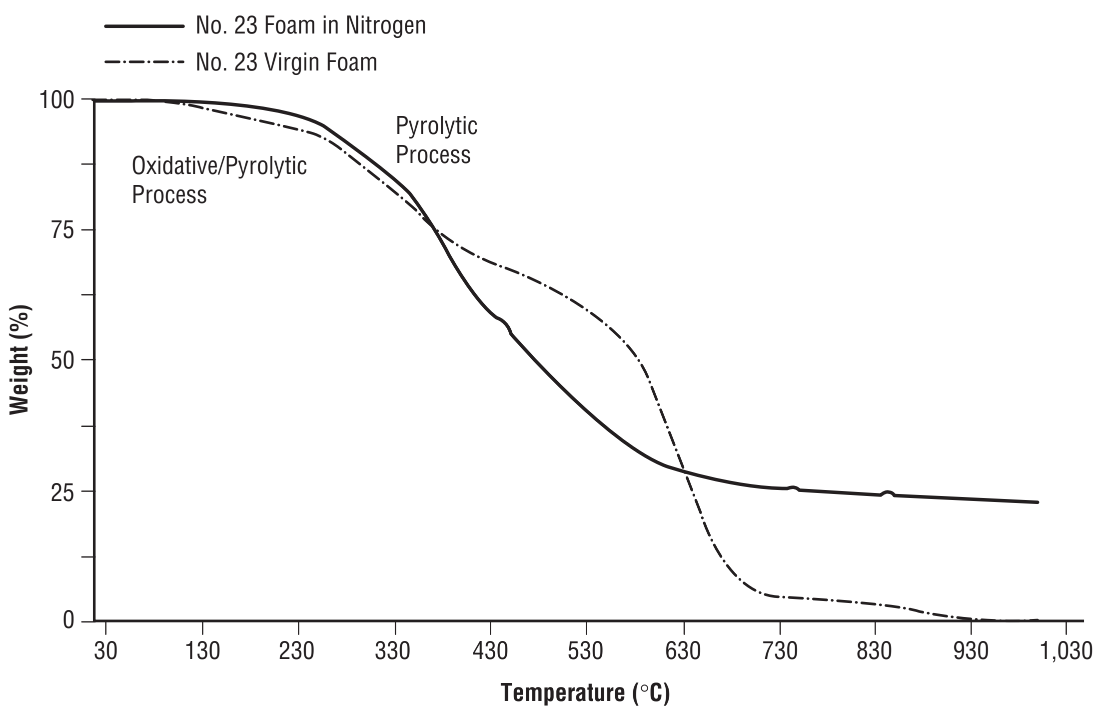

Figure 3. DTA curve of NCFI foam.

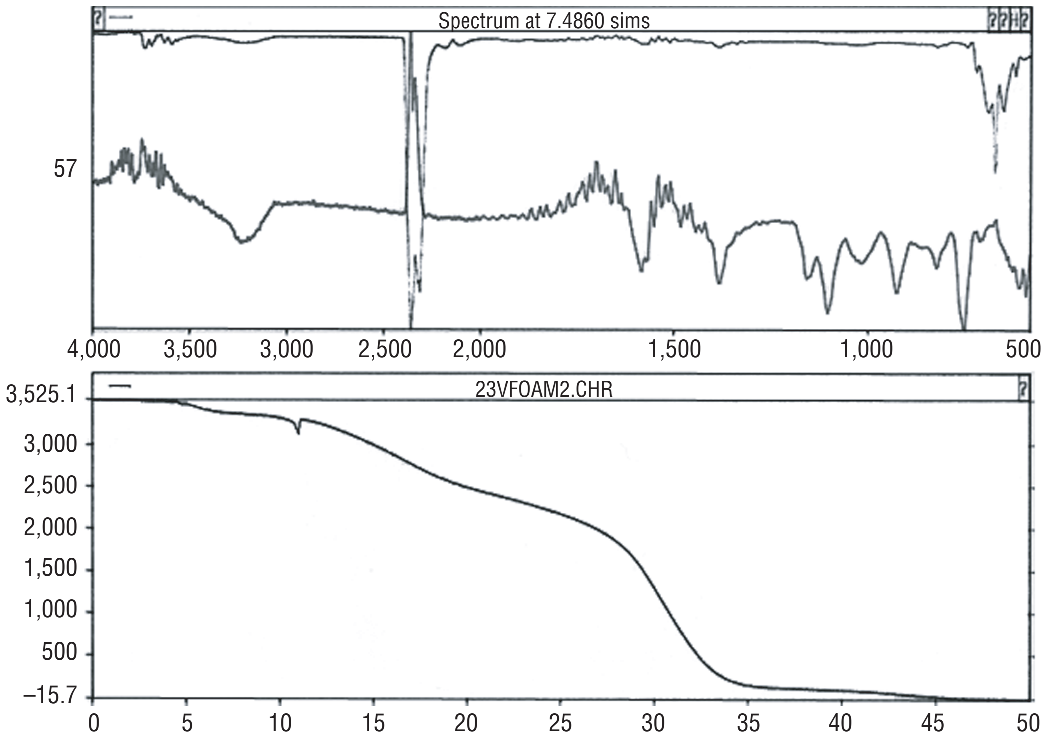

Figure 4. TGA/FTIR spectrometer curve of NCFI foam.

4. ACCOMPLISHMENTS

Results of this research include the development of a comprehensive fingerprint database of composite material behavior under varying conditions of thermal stress. Data include chemical structure characterization, isothermal behavior (crystallization or curing), phase transitions, decomposition (weight loss), heat capacitance, and oxidizing behavior.

The following materials were analyzed for this research effort:

- NCFI foam
- NCFI foam components A and B
- Furon
- TEFZEL®
- Lexan®
- Carbon epoxy fiberglass
- Varglass silicon
- Aluminum alloys
- Aluminum-silicon alloys
- Carbon-graphite seals.

By evaluating the various analytical methods on differing classes of materials, optimization of analytical testing requirements for specific material types was achieved. Based on this research, the order of thermal testing was refined to allow determination of starting temperatures for the thermal analysis tests. It was determined that it is beneficial to analyze nonmetallic materials utilizing the TGA/FTIR spectrometer followed by the DSC or DTA. Based on the decomposition temperatures of the material obtained by the TGA, inferences may be made regarding other thermal properties of the material, including appropriate methodologies. For instance, low decomposition temperatures would suggest equally low or lower temperatures for other thermal properties, such as melting point, heat of fusion, glass transitions, etc. Thus, the DSC, which can operate at subambient temperatures, would make it a more suitable choice for additional analysis. Conversely, the DTA can operate at a much higher temperature than the DSC, suggesting its use when TGA data indicate high-temperature material properties. Other factors must also be weighed for any sample evaluation, including any specific test data objectives sought. For example, if specific heat data are specified, the DSC would be the preferential methodology employed due to its increased sensitivity in heat energy measurements. If chemical characterization is also a requirement, FTIR and/or TGA/FTIR spectroscopy would be appropriate. Nonetheless, based on the starting temperature information garnered from the TGA, the engineer can adequately determine which test(s) is best suited to provide additional material data.

5. PLANNED FUTURE WORK

The investigators will continue to characterize materials received for analysis using the above instrumentation. The investigators will also post the data obtained from this research on the Internet; the site layout has been developed.

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13. ABSTRACT *(Maximum 200 words)*

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